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XXII.

ON A NEW METHOD FOR THE SEPARATION AND SUBSEQUENT TREATMENT OF PRECIPITATES IN CHEMICAL ANALYSIS.

BY F. A. GOOCH.

Presented Feb. 13, 1878.

THE introduction of Bunsen's method of filtration and immediate ignition of precipitates in the moist condition has left little to be desired as regards accuracy of result and rapidity of execution, in the treatment of precipitates which may be submitted to high temperatures in contact with carbonaceous matter. In analytical methods which require that filter-paper and precipitate shall be ignited apart, or dried together at a temperature below the point at which paper begins to char, the same degree of exactness has not, in general, been hitherto attained.

To obviate the difficulty of bringing a paper-filter of ordinary dimensions, particularly when covered with a voluminous precipitate, to a definite condition of desiccation, the sand-filters of Dr. Gibbs and Taylor,* the porous cones of Munroe,† and finally the process of reverse filtering, first applied to quantitative work by Carmichael,‡ improved by Casamajor,§ and thoroughly elaborated by Professor Cooke,‖ have been successively brought forward.

The latter process gives most excellent results in the separation of precipitates which settle quickly and completely; and, inasmuch as many precipitates which of themselves are not inclined to fall rapidly may be made to do so by proper treatment (by boiling, for example, the addition of ammonium salts, Chatard's ¶ method of granulating gelatinous precipitates, or other similar device), the field of usefulness of this process is wide.

The sand-filters in skilful hands, and the porous cones with no more than ordinary care, give accurate results, and possess moreover the advantage of being applicable to the filtration of liquids which quickly destroy woody fibre. The length of time required to dry them thoroughly, and their sensitiveness to atmospheric conditions, are their great drawbacks.

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* Am. Jour. [2], vol. xliv. p. 215.

† Am. Jour. [3], vol. i. p. 1.

‡ Zeitschr. f. Chem. [N.F.], Bd. 6, p. 481.

§ Am. Chem. vol. v. p. 441.

‖ These Proceedings, vol. xii. p. 124.

¶ Am. Jour. [2], vol. l. p. 247.

For the simple filtration of corrosive liquids without reference to a subsequent estimation of the precipitate, filters of sand, broken glass, garnets, and fibrous asbestus have for a long time been used. More recently, asbestus has been moulded into the shape of an ordinary filter: in the dry state by Löwe,* by rubbing between hollow and a solid wooden cones; in the wet state by Gruner,† by grinding asbestus, mixed in a mortar with water, to a pulp, transferring the mixture to a funnel choked with asbestus, inserting an accurately fitting cone of brass gauze, which presses the asbestus against the walls of the funnel, pouring off the water, carefully removing the cone and drying the layer of asbestus which adheres to the glass. Böttger ‡ has used filters of gun-cotton; and Bunsen § has devised a filtering apparatus for corrosive liquids to be attached to his pump, which consists of a disc of artificial pumice fitted to a conical tube and packed around its edge with fibrous asbestus.

None of these later-mentioned methods, however, are well adapted to the quantitative estimation of precipitates.

Impressed with the desirability of further improvement in those processes of quantitative analysis which involve the use of dried filters, or the separation of filter and precipitate before ignition, I have had the good fortune, in taking the matter up in turn, to succeed in devising and preparing a felt of anhydrous asbestus, which is capable of filtering liquids with a rapidity and efficiency at least as great as may be obtained by the use of good filter paper; is light, compact, incombustible at the highest temperatures used in analytical processes; is not acted upon by acids (excepting hydrofluoric acid) or alkalies; is sufficiently coherent to resist entirely the disintegrating action of a liquid forced through it under the pressure of the Bunsen pump, and which may moreover be prepared by a very simple process: in short, a filtering material which, in my belief, makes it possible to reach a high degree of accuracy in many analytical processes which hitherto have been none of the best, and to add to those already known new methods which previously have been impracticable.

My mode of preparing and using the asbestus felt is as follows: —

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* Dingl. pol. Jour. cxlviii. 444.

† Jahresb. Chem. 1869, p. 990.

‡ Dingl. pol. Jour. clv. p. 463.

§ Ann. Ch. Pharm. cxlviii. p. 290.

First, white, silky, anhydrous asbestus is scraped to a fine short down with an ordinary knife-blade, boiled with hydrochloric acid to remove traces of iron or other soluble matter, washed by decantation and set aside for use.

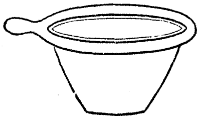

Fig. 1.

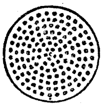

Fig. 2.

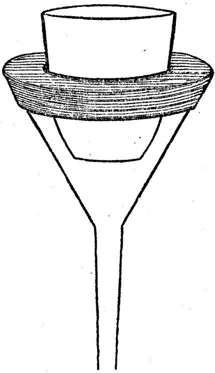

Fig. 3.

Secondly, a platinum crucible of ordinary size, preferably of the broad low pattern (Fig. 1), is chosen, and the bottom (Fig. 2) perforated with fine holes (the more numerous and the finer, the better) by means of a steel point; or, better still, the bottom may be made of fine platinum gauze. Next, a Bunsen funnel of the proper size is selected, and — following Munroe's plan for holding his porous cones — over the top a short piece of rubber tubing is stretched and drawn down until the portion above the funnel arranges itself at right angles to the direction of the stem. Within the opening in the rubber, the perforated crucible is fitted as shown in figure 3, and the funnel is connected with the receiver of a Bunsen pump or other exhausting apparatus in the ordinary manner.

To make the asbestus felt, the pressure of the pump is applied, and a little of the asbestus prepared as described, and suspended in water, is poured into the crucible. The rubber and the crucible are held together by the pressure of the vacuum-pump with sufficient force to make an air-tight joint; the water is drawn through, and the asbestus is deposited almost instantly in a close compact layer upon the perforated bottom; more asbestus (if necessary) in suspension as before being poured upon the first, until the layer becomes sufficiently thick for the purpose for which it is intended. Finally, a little distilled water is drawn through the apparatus to wash away any filaments that might cling to the under side, and the filter is ready for use: the whole process occupying less time than is necessary to fold and fit an ordinary paper-filter to a funnel.

To prepare the filter for the estimation of a precipitate, the crucible with the felt undisturbed is removed from the funnel and ignited. In case the precipitate, to be subsequently collected, must be heated to a very high temperature for a long time, it is better to enclose the perforated crucible with its felt within another crucible; because, in such cases, asbestus felt is apt to curl at the edges, and without such precaution some of the precipitate might drop through the perforations and be lost. For drying at low temperatures, however, and even for ordinary ignitions, a second crucible is unnecessary; but, during the ignition of an easily reducible substance, care must be taken to prevent the contact of unburnt gas with the perforated bottom.

To perform the filtration, the crucible is replaced in the funnel, the pressure applied, and the process conducted precisely as in an ordinary filtration by the Bunsen pump. It is necessary to observe that the vacuum-pump is to be started before pouring the liquid upon the filter. The final drying or ignition, as the case may be, of precipitate and filter is made without difficulty, or need of extra precaution.

When turbid liquids are to be filtered, or gelatinous precipitates to be separated, instead of the perforated crucible, I prefer to use a platinum cone (Figs. 4 and 5), the upper part of foil (to make a tight joint with the rubber fitting of the funnel), the lower of gauze. The method of covering the gauze with felt is identical with that described above. By reason of the larger filtering surface of this apparatus, the tendency to become clogged is, of course, very much diminished. When subjected to prolonged ignition, the gauze cone is enclosed within a crucible or a cone of platinum foil.

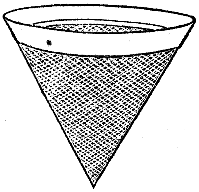
Fig. 4.

In operations in which platinum is liable to receive injury, a porcelain crucible, with a perforated bottom, may be used; but recourse to this is rarely necessary, particularly when one may use the gauze cone protected, as it is, by asbestus felt; moreover, the perforation of porcelain with numerous fine holes is a matter of considerable difficulty and expense.

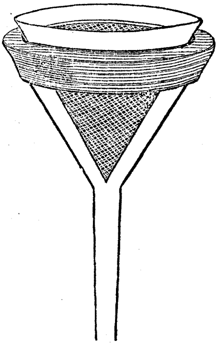
Fig. 5.

Asbestus felt may be also used in the process of reverse filtering, it being merely necessary to dip the platinum rose into the asbestus mixture, after starting the vacuum-pump, in order to make the felt. The rose, with the felt attached, and the vessel in which the precipitate is collected, are to be weighed together, both before and after filtration.

Nothing can be simpler than the whole method of preparation and use of the apparatus which I have described, and its efficiency is extremely great. Clean water may, under the pressure of a Bunsen pump, be passed through a gauze cone coated with asbestus felt, which exposes a filtering surface of twenty-four square centimetres (nearly the same as that of a paper-filter, eight centimetres in diameter, when folded in the ordinary manner), with ease at the rate of a litre per minute.

When the filtering surface is less, the rapidity of filtration is, of course, somewhat diminished, but always exceeds that of paper of the same dimensions. When the felt is deposited upon gauze, the layer may be surprisingly thin and yet be efficient enough for all ordinary purposes. If the layer of felt be quite thick, the filtrate from baric sulphate freshly precipitated in the cold, may be made to pass through clear. But the great superiority of asbestus felt lies in its constancy of weight, whether dried at high or low temperatures, the rapidity with which it may be safely and completely dried, and its refractoriness as regards acids (excepting hydrofluoric acid) and alkalies. These advantages appear most prominently in processes which involve the separation and desiccation: —

First, of precipitates which (like K_2PtCl_6, $[Hg_2]Cl_2$) must be dried at low temperatures, on account of a tendency to decompose spontaneously at high temperatures;

Secondly, of precipitates which (like Sb_2S_3, $PbSO_4$, $ZnCO_3$) have usually been dried slowly and tediously at low temperatures, or have been separated from the filter before ignition, to avoid the danger of reduction by heating in contact with carbon;

Thirdly, of precipitates which (like $(NH_4)_2Mg_2P_2O_8$, $(NH_4)_2Mn_2P_2O_8$) may be ignited in contact with carbon, but which make its complete combustion difficult.

In all cases, the time required to dry or ignite precipitate and filter is a minimum, inasmuch as heat may be applied as soon and to as high a degree as the precipitate itself will permit. Even in processes in which paper filters find their most convenient application, paper has generally no superiority over asbestus felt.

To illustrate the cohesiveness and refractory nature of the felt, I append the account of a single experiment. All of the liquids used had been previously filtered, excepting the distilled water with which the filter was washed, after the passage of each liquid. This precaution was considered unnecessary in the case of the water; but, if it had been taken, the slight *increase* of weight which was observed during the experiment would probably have been prevented.

Weight, in grammes, of crucible and felt, after ignition	20.2020
after second ignition	20.2020
after passing 100 cm^3 of strong HCl through the filter *ten* times, washing and igniting	20.2020
after passing 100 cm^3 of strong HNO_3 through the filter *ten* times, washing and igniting	20.2021
after passing 100 cm^3 of strong H_2SO_4 through the filter *five* times, washing and igniting	20.2022
after passing 100 cm^3 of a mixture of strong H_2SO_4 and water in equal parts, *ten* times through the filter, washing and igniting	20.2024
after passing 100 cm^3 of water, containing in solution 50 grammes of $NaOH$ *ten* times through the filter, washing and igniting	20.2028
Weight of crucible	20.1932
„ „ felt	.0088

The whole process, involving, in all, the filtration of five litres of liquid (of which one litre was viscous), more than forty changes of the receiver, together with the ignition, cooling, and weighing of the crucible and felt seven times, was completed in two hours and ten minutes. The disc of felt was eleven millimetres in diameter.